



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the application of

Daisuke Kanenari et al.

Group Art Unit: 1773

Serial No. 09/901,074

Examiner: Kevin M Bernat

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For: Rubber Laminate and Pneumatic Tire Using The Same

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DECLARATION UNDER RULE 132

Honorable Commissioner of Patent and Trademarks Washington,
D. C. 20231

Sir:

I, Kanenari Daisuke, a citizen of Japan, residing at
c/o THE YOKOHAMA RUBBER CO., LTD. Hiratsuka Factory, 2-1,
Oiwake, Hiratsuka-shi, Kanagawa, Japan, respectfully,
sincerely and solemnly declare:

That I am by profession a chemical engineer and that I
graduated from Yamagata University, Faculty of Engineering,
Department of Applied Chemistry on March, 1992 and finished
the Master Course of Yamagata University, Department of
Engineering Research on March, 1994.

That since April, 1994, I have been employed by the
YOKOHAMA RUBBER CO., LTD. and have been engaged in research
mainly into the development of new tire material, at Second
Research Group since April, 1994, at Forth Research Group
since July, 1998, at First Research Group since June, 1999,
and at Third Research Group since May, 2001, and have been
engaged in research and development of new rubber and resin
materials at Advanced Materials Laboratory Research &
Development Center of THE YOKOHAMA RUBBER CO., LTD.

That I am one of the joint inventors of the invention as disclosed and claimed in the above-mentioned application (i.e., the present application).

In order to show the difference of the adhesive effects between the present invention and the citation inventions (i.e., JP-8-269241 and JP-9-143306), the following Experiments were carried out under my direction and supervision.

Experiments

1. Mixing Conditions

Mixing of Formulations A1 and A2

Rubber was wound around an 8-inch test roll and kneaded, then the compounding agents were added and the result fully mixed to obtain Formulations A1 and A2.

Mixing of Formulations B and C1, C2, and C3

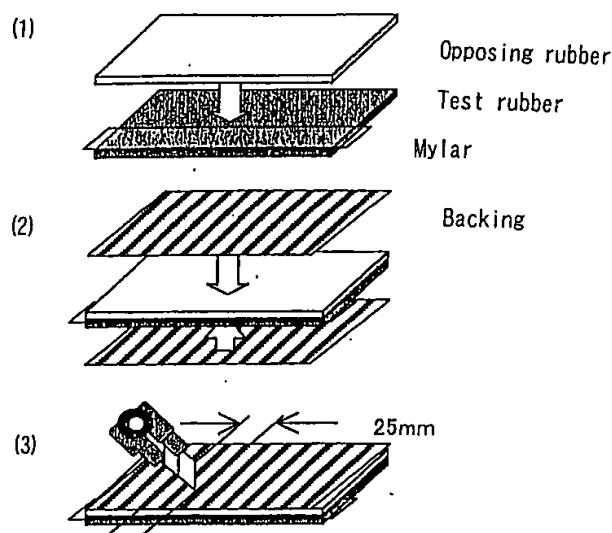
Rubber was charged into a 1.7 liter Banbury mixer (temperature adjusting condition: 60C°) and kneaded for 1 minute, then the compounding agents other than the vulcanization agent were charged. The result was mixed for a total of 5 minutes, then discharged to obtain a master batch. Next, the master batch was wrapped around an 8-inch test roll, the vulcanization agent was charged, then the result was fully mixed to obtain Formulations B, C1, C2, and C3.

2. Method of Preparation of Peeling Test Sample

(1) The formulations were rolled to sheets of a thickness of 2.3 mm by an 8-inch test roll. These were cut to sizes of 15 cm x 15 cm. Rubber sheets to be bonded were laminated with 5 cm width Mylar films (PET treated for separation) sandwiched at the ends.

(2) Next, the two surfaces of the rubber laminates were backed by backing sheets of a thickness of 1 mm comprised of DIP treated polyester cords covered with unvulcanized rubber. The direction of the cords was made 90 degrees with respect to the Mylar film.

(3) The laminates were press vulcanized at 160C° x 30 minutes, then the sheets were cut to widths of 25 mm in the cord direction to obtain peeling samples. The parts sandwiching the Mylar films were gripped for the test.



3. Peeling Test Conditions

1. The bonding test was conducted in accordance with JIS K6256 at a peeling speed of 50 mm/min. The peeling force (N) observed was divided by the sample width to estimate the peeling strength (N/mm).

2. The formulations of Japanese Unexamined Patent Publication (Kokai) No. 8-269241, Japanese Unexamined Patent Publication (Kokai) No.9-143306, and the present invention are shown in the following tables.

Table 1. Hydrogenated NBR Rubber Composition

Name of material	Name of product	Maker	Formulat ion A1 ^{*1}	Formulat ion A2 ^{*2}
Hydrogenated NBR/ZnMA Formulation VnSBR (St=14wt%, Vn=66.2wt%)	ZSC 2295	Nippon Zeon	30	100
Zinc oxide	Zinc White #3	Nippon Zeon	70	
Covulcanizer (TMPT)	Acrylester TMP	Seido Chemical	5	5
Stearic acid	Beads Stearic Acid	NOC	3	
		Kao		0.5
Organic peroxide	Perkadox 14/40	Kayaku Akzo	3	5

Formulation A1^{*1}: Ex. 2 of JP-8-269241

Formulation A2^{*2}: Formulation (A) of JP-9-143306

Table 2. Adhesive Rubber Composition

Name of material	Name of product	Maker	Formulat ion C1 ^{*1}	Formulat ion C2 ^{*2}	Formulat ion C3 ^{*3}
Natural Rubber	RSS #3			60	60
SBR	Nipol 1502	Nippon Zeon	75		
NBR	Nipol DN 401	Nippon Zeon	25	40	
Hydrogenated NBR	Zetpol 2030L	Nippon Zeon			40
Zinc Methacrylate	R-20S	Asada Chemical		10	30
Carbon black	Seast N	Tokai Carbon	50		
Carbon black	Seast 300	Tokai Carbon		50	30
Zinc oxide	Zinc white #3	Seido Chemical	5	5	5
Stearic acid	Beads Stearic Acid	Kao	1	1.5	1.5
Antioxidant	Nocrac 6C	Ouchishinko Chemical		2	2
Aromatic Petroleum resin	FR-120	Fuji Kosan	16	30	
Coumarone resin	Soft coumarone 25B	Kobe Oil Chemical Ind.	16		
Sulfur	Oil treated sulfur	Karuizawa Refinery	2		
Vulcanization accelerator CZ	Nocceler CZ	Ouchishinko Chemical	1		
Organic peroxide	Perkadox 14/40	Kayaku Akzo		3	3
Covulcanizer	DAP monomer	Daiso			15

Formulation C1^{*1}: Ex. 8 of JP-9-143306

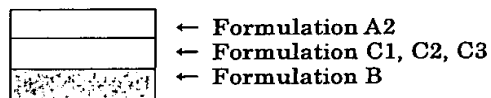
Formulation C2^{*2}: Comp. Ex.1 of Present Invention

Formulation C3^{*3}: Ex. 3 of Present Invention

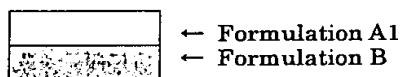
Table 3. Surfur Vulcanized Rubber Composition

Name of material	Name of product	Maker	Formulat ion B
Natural Rubber	RSS #3		50
Polybutadiene rubber	Nipol BR-1220	Nippon Zeon	20
Styrene-butadiene rubber	Nipol 1502	Nippon Zeon	30
Carbon black	Diablack E	Chubu Carbon	60
Zinc oxide	Zinc White #3	Seido Chemical	5
Stearic acid	Beads Stearic Acid	Kao	1
Aromatic oil	Extract No. 4S	Showa Shell Oil	10
Antioxidant	Nocrac 224	Ouchishinko Chemical Ind.	1
Sulfur	Crystex HSOT20	Flexsys	3
Vulcanization accelerator	Nocceler NS-F	Ouchishinko Chemical Ind.	1.5

Lamination Method



JP-9-143306, Present Invention



JP-8-269241

4. Test Results

Test results are shown in the following Tables.

(Note that the unit of peeling strength of Table 1 for Japanese Unexamined Patent Publication (Kokai) No. 8-269241 is N/cm, but in view of the current measured values and the unit of FIG. 1, this is believed to be perhaps a mistake for N/inch (25mm).)

Table 4. Results of Peeling Test of Formulation A (Hydrogenated NBR Composition) and Rubber of Examples of Each Invention

Formulation A	Test rubber		Formulation A2	Formulation A1	Formulation A2	Formulation A2	Formulation A2
Formulation C1, C2, C3	Combined rubber		-	-	Formulation C1	Formulation C2	Formulation C3
Formulation B	Combined rubber		Formulation B	Formulation B	-	-	-
	23°C	N/mm	0.5	5.1	27.6	24.0	15.2
	Peeling state	4-stage judgment	P	G	VG	VG	VG
	120°C	N/mm	0.3	0.8	2.2	3.2	5.6
	Peeling state	4-stage judgment	P	P	P	P	G

Corresponding
Example

JP-8-269241, JP-8-269241, JP-9-143306, Invention, Invention,
Comp. Ex. 5, Ex. 2 Ex. 8 Comp. Ex. 1 Ex. 3
JP-9-143306
Comp. Ex. 2

Table 5. Results of Peeling Test of Formulation B (Sulfur Vulcanized Rubber Composition) and Rubber of Examples of Each Invention

Formulation A	Test rubber		Formulation C1	Formulation C2	Formulation C3
Formulation C1, C2, C3	Combined rubber		Formulation C1	Formulation C2	Formulation C3
Formulation B	Combined rubber		Formulation B	Formulation B	Formulation B
	23°C	N/mm	31.2	30.5	28.6
	Peeling state	4-stage judgment	VG	VG	VG
	120°C	N/mm	18.3	17.4	19.0
	Peeling state	4-stage judgment	VG	VG	VG

Corresponding
Example

JP-9-143306, Invention, Invention,
Ex. 10 Comp. Ex. 1 Ex. 3

* The judgment criteria for the state of peeling are based on the explanation in the specification of the present invention.

VG (very good): Material destruction of rubber with none at all at bonded interface

G (good): Very slight interfacial, but substantially material destruction

F (fair): Interfacial peeling and material destruction mixed

P (poor): Complete interfacial peeling

5. Considerations

As clear from Table 4, the peeling strength of the best Example 2 of Japanese Unexamined Patent Publication (Kokai) No. 8-269241 was lower compared with Example 8 of Japanese Unexamined patent Publication (Kokai) No. 9-143306 and Example 3 of the present invention. Further, the method of Japanese Unexamined Patent Publication (Kokai) No. 8-269241 blends VnSBR in a hydrogenated NBR composition, so the formulations of the high strength hydrogenated NBR compositions end up being limited and therefore this is not practical.

The best Example 8 of Japanese Unexamined Patent Publication (Kokai) No. 9-143306 was high in strength at room temperature (23C°) and higher than Example 3 of the present invention, but in a high temperature (120C°) peeling test, the strength dropped sharply and interfacial peeling occurred. As opposed to this, the formulation of Example 3 of the present invention resulted in destruction of the rubber material even at a high temperature. It was learned that the formulation of the present invention is particularly superior in bonding strength at a high temperature.

The bondability (Table 5) with respect to a sulfur-vulcanized diene-based rubber composition (Formulation B) is of a sufficient level both at room temperature and high

temperature.

As clear from these results, the formulations of the present invention enable improvement of the high temperature bonding and improvement of the durability at the running time of the run-flat tire when used for side reinforcement rubber of a run-flat tire. Therefore, it is clear that the present invention has inventive step with respect to Japanese Unexamined Patent Publication (Kokai) Nos. 8-269241 and 9-143306.

I, the undersigned declarant, declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and; further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001, of Title 18, of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed this 17th day of December, 2003

Shisuke Kanenari